

Patent Claims:

- 5 *Sub A1*
1. A process for the preparation of a heat-stable
antimony-free polyester of neutral color by
esterification of an aromatic dicarboxylic acid or
transesterification of a lower aliphatic ester of
an aromatic dicarboxylic acid with an aliphatic
diol and subsequent polycondensation, which
comprises a procedure in which
10 a possible transesterification is carried out in
the presence of 20 to 120 ppm, based on the
catalyst metal, of a transesterification catalyst,
then
15 after the esterification or transesterification
has ended,
phosphoric acid, phosphorous acid and/or a
phosphonic acid or a derivative thereof are added
to the esterification or transesterification batch
as a complexing agent in an amount which is 100%
20 of the amount equivalent to the
transesterification catalyst employed and up to
99% of the amount equivalent to the cobalt to be
employed,
up to 80 ppm of cobalt in the form of a cobalt
25 compound are added to the batch,
and the polycondensation is carried out without
the addition of antimony, in the presence of 1 to
10 ppm of titanium, which is added in the form of
a titanium compound, and if appropriate in the
30 presence of up to 1000 ppm of organic compounds
which donate crosslinking structural groups
(pentaerythritol)
and if appropriate up to 50 ppm of an optical
35 brightener.
2. The process as claimed in claim 1, wherein, after
the esterification or transesterification has
ended,
phosphoric acid, phosphorous acid and/or a phos-

phonic acid or a derivative thereof is added as the complexing agent to the esterification or transesterification batch in an amount of 100% of the amount equivalent to the transesterification catalyst employed and 90 to 99% of the amount equivalent to the cobalt to be employed.

3. The process as claimed in claim 1, wherein the polycondensation is carried out without the addition of antimony in the presence of 1 to 10 ppm of titanium up to an IV, measured in dichloroacetic acid at 25°C, of 0.4 to 0.9 dl/g and up to a carboxyl group concentration of 10 to 50 mmol/kg in the melt, and then up to the desired end viscosity in the solid phase.

4. The process as claimed in claim 1, wherein 20 to 40 ppm of cobalt in the form of a cobalt compound are added to the batch.

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5. The process as claimed in claim 1, wherein the polycondensation is carried out without addition of antimony in the presence of 2-8 ppm of titanium and if appropriate in the presence of up to 1000 ppm of organic compounds which donate crosslinking structural groups.

6. The process as claimed in claim 1, wherein the polycondensation is carried out in the presence of 100 to 500 ppm of organic ^{crosslinking agents} ~~compounds which donate crosslinking structural groups.~~

7. The process as claimed in claim 1, wherein the polycondensation is carried out ~~without addition of antimony~~ in the presence of up to 25 ppm of an optical brightener.

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8. The process as claimed in claim 1 by esterification of an aromatic dicarboxylic acid or hydroxycarboxylic acid or transesterification of a lower aliphatic ester of an aromatic dicarboxylic acid or hydroxycarboxylic acid with an aliphatic diol and subsequent polycondensation, wherein 80 to 100 mol% of an aromatic dicarboxylic acid of the formula III



or a lower aliphatic ester thereof and 0 to 20 mol% of an aromatic hydroxycarboxylic acid of the formula IV



or a lower aliphatic ester thereof, are esterified or transesterified with a diol of the formula V



in which

X is, based on the total amount of di- and hydroxycarboxylic acids, aromatic radicals having 5 to 16, preferably 6 to 12 carbon atoms to the extent of more than 80 mol%, and not more than 20 mol% of aliphatic radicals having 4 to 10 carbon atoms, preferably 6 to 8 carbon atoms,

X¹ is the p-phenylene radical,

Y is, based on the total amount of transesterified or esterified diols, alkylene or polymethylene groups having 2 to 4 carbon atoms or cycloalkane or dimethylene-cycloalkane groups having 6 to 10 carbon atoms to the extent of at least 80 mol% and straight-chain or branched alkanediyl having 4 to 16, preferably 4 to 8, carbon atoms or radicals of the formula $-(\text{C}_2\text{H}_4\text{-O})_n\text{-C}_2\text{H}_4\text{-}$, in which n is an integer from 1 to 40, to the extent of not more than 20 mol%, where n is preferably 1 or 2 for contents up to 20 mol%, and groups where n = 10 to 40 are preferably present only in contents of less than 5 mol%.

9. The process as claimed in claim 8, wherein X is, based on the total amount of di- and hydroxycarboxylic acids, p-phenylene radicals to the extent of 90 to 100 mol%, m-phenylene radicals to the extent of 0 to 7 mol% and aliphatic radicals having 4 to 10 carbon atoms, preferably 6 to 8 carbon atoms, to the extent of 0 to 5 mol%, X^1 is the p-phenylene radical, Y is, based on the total amount of transesterified or esterified diols, alkylene or polymethylene groups having 2 to 4 carbon atoms or cycloalkane or dimethylene-cycloalkane groups having 6 to 10 carbon atoms to the extent of at least 90 mol% and straight-chain or branched alkanediyl having 4 to 16, preferably 4 to 8, carbon atoms or radicals of the formula $-(C_2H_4-O)_n-C_2H_4-$, in which n is the number 1 or 2, to the extent of not more than 10 mol%.

10. A heat-stable, antimony-free polyester of neutral color based on an aromatic dicarboxylic acid and an aliphatic diol, ~~which can be prepared by the process as claimed in claim 1,~~ in which, in the non-matted state, its color number components are a^* in the range from -3 to +3, b^* in the range from -6 to +6 and L^* in the range from 55 to 75.

11. A heat-stable, antimony-free polyester of neutral color based on an aromatic dicarboxylic acid and an aliphatic diol as claimed in claim 10, which is free from antimony, and comprises 1 to 10 ppm of titanium, 20 to 120 ppm of a transesterification catalyst metal in the form of catalytically inactive complexes with phosphoric acid, phosphorous acid and/or a phosphonic acid or a derivative thereof, and 0 to 80 ppm of cobalt, which is partly present in the form of catalytically inactive complexes

with phosphoric acid, phosphorous acid and/or a phosphonic acid or a derivative thereof, and optionally up to 50 ppm of an optical brightener.

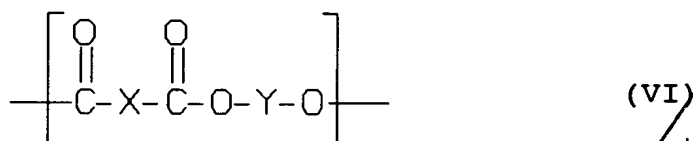
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12. A heat-stable, antimony-free polyester of neutral color as claimed in claim 10, which is free from antimony and comprises 2 to 8 ppm of titanium, 50 to 90 ppm of manganese (calculated as the metal) in the form of catalytically inactive complexes with phosphoric acid, phosphorous acid and/or a phosphonic acid or a derivative thereof, and 20 to 40 ppm of cobalt, which is partly in the form of catalytically inactive complexes with phosphoric acid, phosphorous acid and/or a phosphonic acid or a derivative thereof, and optionally up to 25 ppm of an optical brightener.

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13. A heat-stable, antimony-free polyester of neutral color as claimed in claim 10, in which, in the non-matted state, its color number components are a* in the range from -2 to +2, b* in the range from -3.5 to +3.5 and L* in the range from 60 to 70.

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14. A heat-stable, antimony-free polyester of neutral color as claimed in claim 10, in which 90 to 99% of the cobalt is in the form of one or more catalytically inactive complexes with phosphoric acid, phosphorous acid and/or a phosphonic acid or a derivative thereof.

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15. A heat-stable, antimony-free polyester of neutral color as claimed in claim 10, which comprises 5 to 25 ppm of an optical brightener.

16. A heat-stable, antimony-free polyester of neutral color as claimed in claim 10, wherein its polymer chains are built up from 80 to 100 mol% of structural groups of the formula VI



and 20 to 0 mol% of structural groups of the formula VII



in which

X is aromatic radicals having 5 to 16, preferably 6 to 12 carbon atoms to the extent of more than 80 mol% and not more than 20 mol% of aliphatic radicals having 4 to 10 carbon atoms, preferably 6 to 8 carbon atoms,
X¹ is the p-phenylene radical,
Y is alkylene or polymethylene groups having 2 to 4 carbon atoms or cycloalkane or dimethylene-cycloalkane groups having 6 to 10 carbon atoms to the extent of at least 80 mol% and straight-chain or branched alkanediyl having 4 to 16, preferably 4 to 8 carbon atoms or radicals of the formula $-(\text{C}_2\text{H}_4-\text{O})_n-\text{C}_2\text{H}_4-$, in which n is an integer from 1 to 40, to the extent of not more than 20 mol%, where n is preferably 1 or 2 for contents up to 20 mol% and groups where n = 10 to 40 are preferably present only in contents of less than 5 mol%.

17. A heat-stable, antimony-free polyester of neutral color as claimed in claim 16, which consists of

structural groups of the formula IV in which
X is p-phenylene radicals to the extent of 90 to
100 mol%, m-phenylene radicals to the extent of 0
to 7 mol% and aliphatic radicals having 4 to 10
carbon atoms, preferably 6 to 8 carbon atoms, to
the extent of 0 to 5 mol%,
Y is alkylene or polymethylene groups having 2 to
4 carbon atoms or cycloalkane or dimethylene-
cycloalkane groups having 6 to 10 carbon atoms to
the extent of at least 90 mol% and
straight-chain or branched alkanediyl having 4 to
16, preferably 4 to 8 carbon atoms or radicals of
the formula $-(C_2H_4-O)_n-C_2H_4-$, in which n is the
number 1 or 2, to the extent of not more than
10 mol%.

18. A heat-stable, antimony-free polyester of neutral
color as claimed in claim 16, which is composed of
structural groups of the formula IV in which
X is p-phenylene radicals to the extent of 93 to
99 mol% and m-phenylene radicals to the extent of
1 to 7 mol%.

19. A heat-stable, antimony-free polyester of neutral
color as claimed in claim 10, in which the
catalytically inactive complexes of manganese and
of cobalt are complexes with phosphorous acid or
an ester thereof.

20. A heat-stable, antimony-free polyester of neutral
color as claimed in claim 10, which ^{further} optionally
comprises up to 1000 ppm of crosslinking
structural groups.

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